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EMITTANCE OF TD-NiCr AFTER SIMULATED
REENTRY HEATING

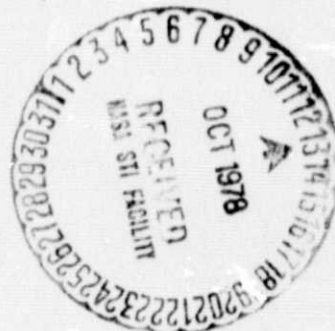
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EMITTANCE OF TD-NiCr AFTER
SIMULATED REENTRY HEATING

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SUMMARY

The effects of simulated reentry heating on the emittance of TD-NiCr were investigated. Groups of specimens were subjected to three different preconditioning treatments. Specimens from each group were exposed to 6, 24, and 30 half-hour simulated reentry exposure cycles in a supersonic arc tunnel at each of three conditions intended to produce surface temperatures of 1255, 1365, and 1475 K. Spectral and total normal emittance were determined at 1300 K on specimens which had been preconditioned only and specimens after completion of reentry simulation exposure. Oxide morphology and chemistry were studied by scanning electron microscopy and X-ray diffraction analysis.

A consistent relationship was established between oxide morphology and total normal emittance. Specimens with coarser textured oxides tended to have lower emittances than specimens

with finer textured oxides. However, no systematic relationship between emittance and either exposure time, exposure temperature, or type of preconditioning was evident. No correlation was found between emittance and surface chemistry.

Specimens which were not exposed to simulated reentry heating had emittances on the low end (0.62-0.70) of the range reported in the literature for statically oxidized TD-NiCr (0.6 to 0.9). Specimens exposed to larger numbers of reentry simulation cycles at 1365 and 1475K had emittances as low as 0.52. Results presented in this paper suggest that emittance values previously assumed in design studies of advanced space transportation systems may be too high.

INTRODUCTION

Advanced space transportation systems and hypersonic aircraft which experience severe aerodynamic heating must possess some means of thermal shielding. One shielding scheme employs a reusable high emittance metallic external surface (heat shield) which reradiates most of the incident energy. Nickel base superalloys have been the focus of considerable interest for heat shield applications because of their high temperature strength and oxidation resistance.

One superalloy which has been the subject of extensive laboratory study over the past ten years is TD-NiCr, a dispersion strengthened nickel-chromium alloy. Technical evaluations of

this alloy have indicated that it may be usable for temperatures up to 1500 K for heat shield applications (refs. 1 and 2). This is 100 K or more above the maximum use temperature of conventional superalloys.

The successful use of TD-NiCr in radiative TPS applications requires that it have a high, stable emittance in addition to being resistant to temperatures of the order of 1500 K. Design calculations of the temperature distribution of TD-NiCr heat shields for advanced space transportation applications have been based on an assumed emittance of 0.8 to 0.9 (refs. 3 and 4). Statically oxidized TD-NiCr is reported to have a total emittance ranging from 0.6 to 0.9 (ref. 3). However, as shown in reference 5, the oxidation behavior of TD-NiCr and other metallic systems is vastly different under dynamic (high velocity air) as opposed to static conditions (quiescent air). Reference 6 shows that the Cr_2O_3 layer produced during static oxidation is not stable during subsequent arc tunnel exposure and is replaced by a porous NiO layer. It further shows that the oxide scale is repeatedly vaporized and reformed with a period of 30 minutes to 2 hours. The presence of different oxide species may affect the emittance and the catalytic behavior of the surface, thus resulting in different equilibrium surface temperatures under the same stream conditions in flowing air. This paper presents results from a test program aimed at defining the effects of surface preconditioning, exposure temperature, and exposure time on the emittance of TD-NiCr subjected to simulated reentry conditions in

an arc tunnel.

PROCEDURE

Three groups of ten specimens were fabricated from 0.64 mm thick TD-NiCr sheet. The specimens were 60-degree circular segments with an area of approximately 14 cm^2 . Each group of specimens was subjected to mechanical/chemical surface treatment followed by ultrasonic cleaning and static oxidation for 12 hours in air at 1365 K. The mechanical and chemical surface treatments are outlined in Table I.

Reentry Simulation Cycles

Specimens from each group were exposed to 6, 24, and 30 half-hour simulated reentry heating cycles in a supersonic arc tunnel at each of three conditions intended to produce target surface temperatures of 1255, 1365, and 1475 K. During arc tunnel testing, the specimens were oriented normal to the flow and were exposed to heating rates of 112 to 216 kw/m^2 , mass flow rates of 4.8 to 6.1 g/s, enthalpies of 7.4 to 13.0 MJ/kg and stagnation pressure of 0.006 atm. Between successive heating cycles, specimens were allowed to cool in air for thirty minutes. These tests were conducted on contract by the Aerotherm Division of Acurex Corporation. Measurements were made of specimen surface temperature, surface recession and mass loss at six cycle intervals. References 7 and 8 present the test conditions and results of these tests.

Emittance Measurements

Spectral normal emittance measurements were made on specimens after preconditioning and on specimens after preconditioning and reentry simulation heating. Measurements were taken at discrete wavelengths over the range from 1 μm to 15 μm at a temperature of 1300 K. Data were taken at 0.5 μm intervals between 1 and 3 μm and at 1 μm intervals between 3 and 15 μm . These data were obtained by the radiometric technique described in reference 9. Total normal emittance (ϵ_{TN}) was calculated by numerical integration of the spectral emittance data.

Laboratory Analysis

The morphology of the oxide scale of each specimen was studied by conventional light microscopy and scanning electron microscopy (SEM) at magnifications up to 1500X. Elemental analysis of the oxide scale was carried out during SEM examination by using an energy dispersive X-ray analysis system (EDAX). The compounds or phases present in the oxide scale were identified by conventional X-ray diffraction analysis employing nickel-filtered copper K_{α} radiation.

RESULTS

A plot of emittance versus wavelength at 1300 K is shown in figure 1. The shape of the spectral emittance distribution is representative of the specimens tested in this program. Different exposure conditions resulted in varying spectral emittance values

at specific wavelengths especially in the 1 to 6 μm region, but the general shape of spectral distribution remained the same. Maximum emittance occurred near 13 μm and minimum emittance occurred near 2 μm .

Table II presents the total normal emittance and the results of X-ray diffraction analysis for each specimen. These data were arranged according to the target surface temperature, number of simulated reentry cycles and type of preconditioning. Specimens with no simulated reentry exposure exhibited total emittances ranging from 0.62 for type A to 0.70 for type C, which is on the low end of the published values of 0.60 to 0.90 for statically oxidized TD-NiCr. Exposure to six simulated reentry cycles at the lower target surface temperature resulted in emittances which were greater than those of the specimens with no simulated reentry exposure. In general, specimens exposed to a greater number of simulated reentry cycles at the lowest target surface temperature exhibited lower emittances than did the specimens exposed to six cycles. The emittances of specimens exposed to the two higher target surface temperatures for large numbers of simulated reentry cycles were noticeably lower (0.50 to 0.60) than those of specimens exposed for six cycles or those of specimens with no simulated reentry exposure and significantly lower than the value of 0.80 to 0.90 used in design studies.

An examination of the X-ray diffraction data in Table II shows that the specimens with no simulated reentry exposure had a

surface consisting of Cr_2O_3 . On the other hand, both NiO and NiCr_2O_4 spinel, but not Cr_2O_3 , were detected on the surfaces of specimens exposed to simulated reentry. NiCr_2O_4 was not detected in every case. On specimens where NiO and NiCr_2O_4 were both detected, their relative intensities were highly variable, but NiO was always the predominant species. The presence of Ni and ThO_2 was also indicated. However, these signals are believed to be the result of X-ray penetration of the oxide scale into the underlying alloy. Overall, little correlation between surface chemistry and emittance was found.

Attempts at correlating emittance data with target surface temperature, exposure time, and specimen preconditioning were not successful. However, a detailed examination of oxide morphology yielded a consistent relationship between the texture of the oxide scale and the total normal emittance. Figures 2 through 5 present SEM photographs with emittance data showing the relation between emittance and morphology as a function of preconditioning type, number of simulated reentry cycles and target surface temperature.

The morphology and total normal emittance resulting from the different preconditioning treatments is shown in figure 2. All three preconditioning treatments resulted in the formation of a continuous oxide scale. EDAX showed the oxide scale to be rich in Cr which is consistent with X-ray diffraction results. However, the specimen with type-A preconditioning had a somewhat coarser texture and lower emittance than the specimens with either type-B

or type-C preconditioning.

Figure 3 shows SEM photographs and total normal emittance of type-A preconditioned specimens after exposure to 6, 24, and 30 simulated reentry cycles at 1475 K. The 6-cycle specimen had an extensive population of small mushroom-shaped oxide clusters embedded in a dark green oxide matrix. EDAX scans indicated that the "mushrooms" were rich in Ni and that the matrix contained more Cr than Ni. The 24-cycle specimen had a population of somewhat larger mushrooms, and the 30-cycle specimen had a heavy growth of very large mushrooms which had coalesced to yield a very coarse texture. Stereo microscope examination of these specimens showed evidence of local spallation of the mushroom layer followed by regrowth of smaller mushrooms on the 24-cycle specimen and fully healed spallation areas on the 30-cycle specimen. The progressive coarsening of the oxide scale with increasing exposure time was accompanied by a decreasing total normal emittance. Although the trend shown in figure 3 is representative of all specimens exposed at the highest target temperature, the longest exposure times at the two lower temperatures did not produce the coarsest textures or lowest emittances among these specimen groups.

The morphology and total normal emittance of specimens subjected to type-B preconditioning and exposed to 24 simulated reentry cycles at the three target surface temperatures are shown in figure 4. The mushrooms on the specimens exposed at 1255 and 1475 K were relatively small and did not completely cover the

underlying oxide. The mushrooms on the specimen exposed to the 1365 K had coalesced and were considerably larger than those on the other two specimens. Likewise, the emittance of the 1365 K specimen is substantially lower than that of the 1475 and 1255 K specimens. The emittances of specimens subjected to type-C preconditioning had a similar relationship to oxide morphology as shown here, in that the 1365 K specimen which had the coarsest texture also had the lowest emittance. However, the 1365 and 1475 K specimens subjected to type-A preconditioning had equal emittances even though the 1365 K specimen had a coarser texture.

Figure 5 presents the morphology and total normal emittance of specimens from each preconditioning group that were exposed to 30 simulated reentry cycles at the highest target mushrooms. Unlike the specimens subjected to type-A and type-B preconditioning, the specimen subjected to type-C preconditioning showed no evidence of mushroom coalescence. Similarly the emittance of the type-C specimen was not so low as that of the other two.

An examination of the emittance results shows no systematic relationship between oxide morphology and exposure temperature, exposure time, or type of preconditioning. However, a reasonably consistent relationship exists between oxide morphology and total normal emittance; that is, coarser textured surfaces generally produced lower emittances than did finer textured surfaces. The repeated vaporization and reformation of the oxide scale described in reference 6, suggests that the development of morphology and, therefore, the emittance may occur in a periodic fashion. Hence,

correlation of morphology with exposure temperature and time may not be possible. The absence of a correlation between morphology/emittance and preconditioning is not surprising in view of the finding in reference 10 that the effects of preconditioning on morphology are limited to relatively short exposure times.

CONCLUDING REMARKS

An investigation was carried out to determine the effect of simulated reentry on the emittance of TD-NiCr. Parameters included in the investigation were: surface preconditioning, exposure temperature, and time of exposure to simulated reentry. The emittance of each specimen was measured and the morphology and composition of the oxide scale on each specimen was studied.

A reasonably consistent relationship between oxide morphology and total normal emittance was established. Specimens with coarser textured oxides tended to have lower emittances than specimens with finer textured oxides.

Wide variations of morphology and emittance were displayed. No systematic relationship between these variations and either number of reentry cycles, target exposure temperature, or type of preconditioning was evident. However, specimens exposed to 24 and 30 simulated reentry cycles at the two higher target surface temperatures tended to have significantly coarser morphology and lower emittance than specimens exposed for only six cycles or specimens which had been preconditioned only. No correlation

between emittance and surface chemistry was found.

The values of emittance on specimens with no simulated reentry exposure were on the low end of the range reported in the literature for statically oxidized TD-NiCr. The emittance of specimens subjected to simulated reentry heating for large numbers of cycles were generally 15 to 20% lower than the emittance of specimens with no simulated reentry exposure. These results suggest that emittance values previously assumed in design studies of advanced space transportation systems may be too high.

REFERENCES

1. Johnson, R., Jr., and Killpatrick, D.H.: Evaluation of Dispersion Strengthened Nickel-Base Alloy Heat Shields for Space Shuttle Application. NASA CR-2614, 1976.
2. Eidinoff, H.L., and Rose, L.: Thermal-Structural Evaluation of TD Ni-20Cr Thermal Protection System Panels. NASA CR-132487, 1974.
3. Johnson, R., Jr., and Killpatrick, D. H.: Evaluation of Dispersion Strengthened Nickel-Base Alloy Heat Shields for Space Shuttle Application. Phase I Summary Report. NASA CR-132360, 1973.
4. Black, W.E., et al: Evaluation of Coated Columbium Alloy Heat Shields for Space Shuttle Thermal Protection System Application. Vol. I, Phase I-Environmental Criteria and Material Characterization. NASA CR-112119, 1972.
5. Johnston, James R., and Ashbook, Richard L.: Oxidation and Thermal Fatigue Cracking of Nickel- and Cobalt Base Alloys in a High Velocity Gas Stream. NASA TN D-5376, 1969.

6. Tenney, D.R.; Young, C.T., and Herring, H.W.: Oxidation Behavior of TD-NiCr in a Dynamic High Temperature Environment. Metallurgical Transactions, Vol. 5, May 1974, pp. 1001-1012.
7. Schaefer, John W.: Thermal Screening of Shuttle Orbiter Vehicle TPS Materials Under Convective Heating Conditions. Vol I, Final Report, NASA CR-114521, 1973.
8. Schaefer, John W.: Thermal Screening of Shuttle Orbiter Vehicle TPS Materials Under Convective Heating Conditions. Vol. II, Tabulation of Test Results. NASA CR-114522, 1973.
9. Edwards, S. Franklin, Kantsios, Andronicos G., Voros, John P., and Stewart, W.F.: Apparatus Description and Data Analysis of a Radiometric Technique for Measurements of Spectral and Total Normal Emittance. NASA TN D-7798, 1975.
10. Young, C.T., Tenney, D.R., and Herring, H.W.: Dynamic Oxidation Behavior of TD-NiCr Alloy with Different Surface Pretreatments. Metallurgical Transactions A, Vol. 6A, Dec. 1975, pp. 2253-2265.

Table I.-Surface Treatment Used in Preconditioning
TD-NiCr Specimens

Precondition- ing Type	Mechanical Treatment	Chemical Treatment
A	80 Mesh SiC Grit Blast	None
B	120 Mesh SiC Grit Blast	Etched in solution of 1 part* HNO_3 , 5 parts HCl and 6 parts Glycerine for 40 min. at 294 K.
C	120 Mesh SiC Grit Blast	Etched in solution of 8 parts HNO_3 , 1 part HF and 16 parts H_2O for 40 min. at 344 K.

* By volume

Table II.-Emittance and X-Ray Diffraction Results of TD-NiCr Subjected to Various Preconditioning Treatments and Reentry Simulation Conditions

Target Surface Temp., K	No. of Simulated Reentry Cycles	Type of Preconditioning	TN	Phases Identified by X-ray Diffraction Analysis				
				Ni	ThO ₂	Cr ₂ O ₃	NiO	NiCr ₂ O ₄
--	--	A	.62	s	vw	vw		
--	--	B	.66	vs	vw	w		
--	--	C	.70	vs	vw	vw		
1255	6	A	.75	vs	vw		m	w
1255	6	B	.76	vs	vw		s	vw
1255	6	C	.76	vs	vw		s	vw
1255	24	A	.62	vs	vw		m	vw
1255	24	B	.61	vs			vs	vw
1255	24	C	.61	s			vs	
1255	30	A	.65	w			vs	vw
1255	30	B	.72	vs	vw		s	
1255	30	C	.65	vs	vw		vs	
1365	6	A	.63	s	vw		vs	vw
1365	6	B	.58	vs	vw		s	w
1365	6	C	.63	s			vs	vw
1365	24	A	.55	w			vs	
1365	24	B	.52	m	vw		vs	
1365	24	C	.57	w	vw		vs	
1365	30	A	.55	m			m	
1365	30	B	.54	vs	vw		vs	
1365	30	C	.57	s	vw		vs	
1475	6	A	.68	vs	vw		m	w
1475	6	B	.67	vs	vw		w	w
1475	6	C	.63	vs	vw		m	w
1475	24	A	.55	w	vw		m	vw
1475	24	B	.63	m	w	m	vw	
1475	24	C	.62	m	vw		s	vw
1475	30	A	.52	w	vw		s	vw
1475	30	B	.52	m	w	s	vw	
1475	30	C	.57	m	vw		s	vw

vs = very strong; s = strong; m = medium; w = weak;
vw = very weak; Blank = not detected.

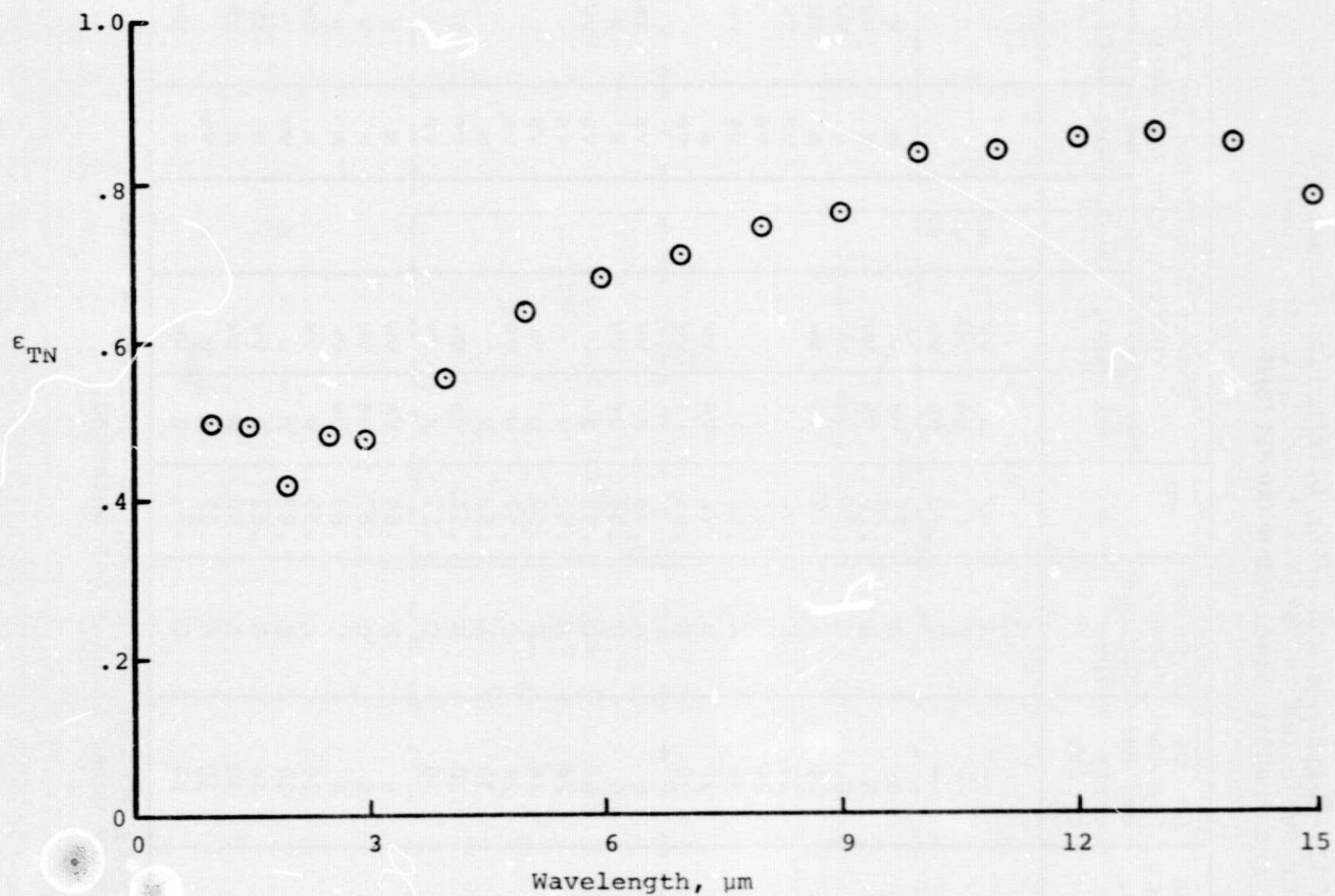
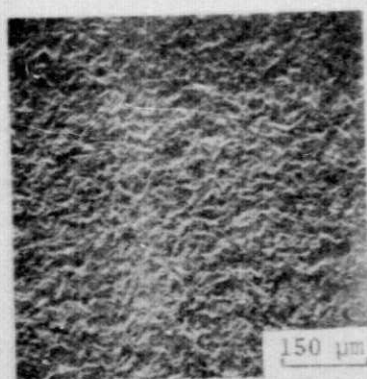
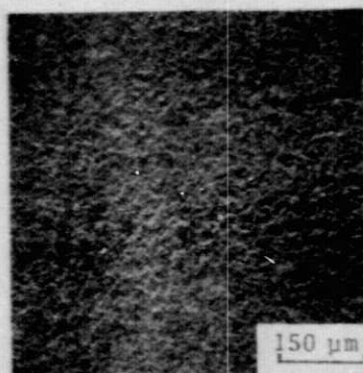


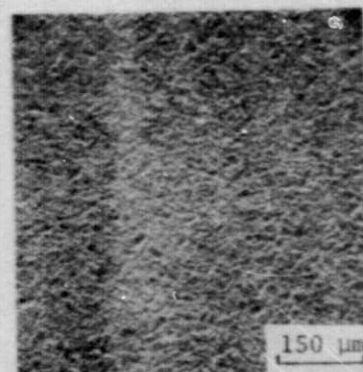
Figure 1.- Spectral Emittance Distribution for TD-NiCr at 1300 K After Type B Preconditioning and 30 Simulated Reentry Cycles at a Target Surface Temperature of 1365 K.



Type A
 $\epsilon_{TN} = 0.62$



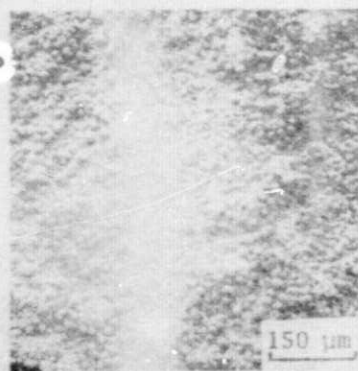
Type B
 $\epsilon_{TN} = 0.66$



Type C
 $\epsilon_{TN} = 0.70$

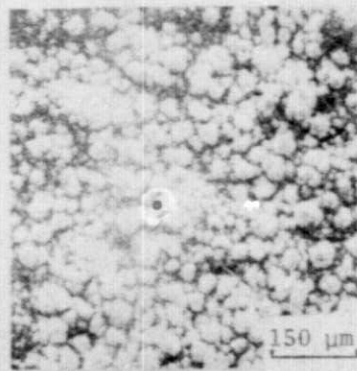
Figure 2.- Morphology and Emittance of TD-NiCr With no Simulated Reentry Cycles.

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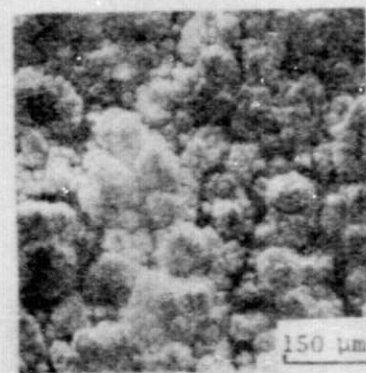
6 Cycles

$$\epsilon_{TN} = 0.68$$



24 Cycles

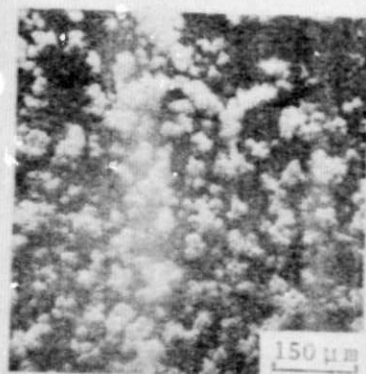
$$\epsilon_{TN} = 0.55$$



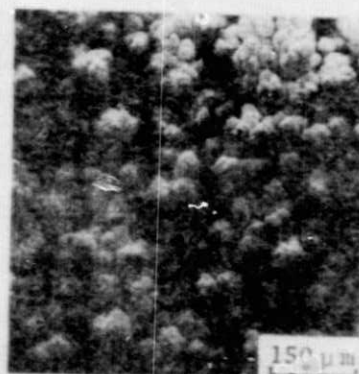
30 Cycles

$$\epsilon_{TN} = 0.52$$

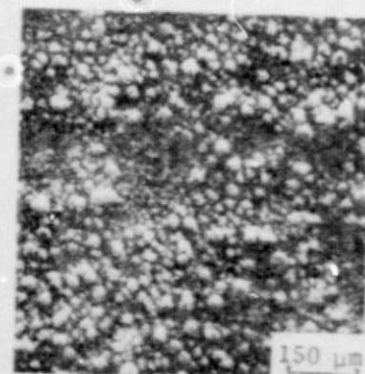
Figure 3.- Morphology and Emittance of TD-NiCr Subjected to Preconditioning Type A and Exposed to 6, 24, and 30 Simulated Reentry Cycles at a Target Surface Temperature of 1475 K.



1255 K
 $\epsilon_{TN} = 0.61$

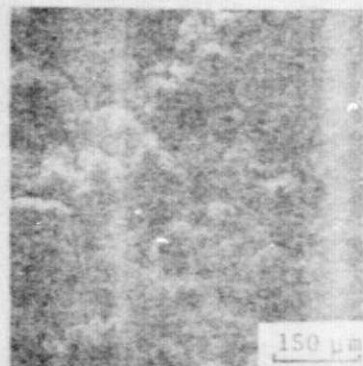


1365 K
 $\epsilon_{TN} = 0.52$



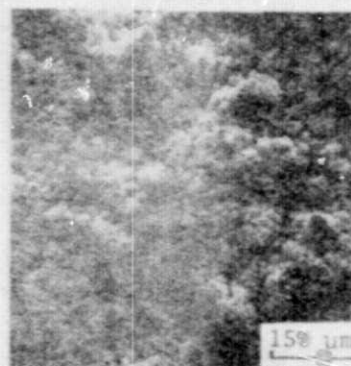
1475 K
 $\epsilon_{TN} = 0.63$

Figure 4.- Morphology and Emittance of TD-NiCr Subjected to Preconditioning Type B and Exposed to 24 Reentry Simulation Cycles at Target Surface Temperatures of 1255, 1365, and 1475 K.



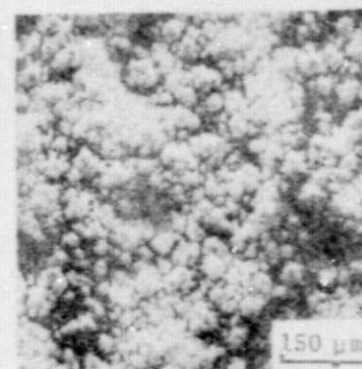
Type A

$$\epsilon_{TN} = 0.52$$



Type B

$$\epsilon_{TN} = 0.52$$



Type C

$$\epsilon_{TN} = 0.57$$

Figure 5.- Morphology and Emittance of TD-NiCr Subjected to Various Types of Preconditioning and Exposed to 36 Reentry Simulation Cycles at a Target Surface Temperature of 1475 K.

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